

Serial number 10/016,844

REMARKS

The applicants and their attorney thank the Examiner for the interview held on 25 January 2005.

During that interview arguments were presented to the Examiner in explanation of the current invention being an adhesive having two separate curing events and distinguishing the current invention from the cited prior art compositions having one curing event. The Examiner suggested a return to the initial claim language for claim one, which has been done by amendment in this response. Reference was made to the Declaration by Kevin Becker submitted with the response to the last office action. The Examiner indicated that the Declaration would be reviewed and considered in this response.

The Examiner also suggested that additional information be presented in the form of DSC profiles for the untested closest prior art examples in Capote and Young. This work has been performed for the Young examples, but not for the Capote, as it was deemed unnecessary after a rereading of the Capote patent.

The Capote invention relates to a resin system, comprising epoxy, cyanate ester, and bismaleimide, which co-cures. In Example 1, Capote discloses the synthesis of 2-allylphenyl glycidyl (APGE) ether; a compound used specifically to co-cure the chemistries: the allyl functionality on the APGE can copolymerize with the bismaleimide, and the glycidyl ether functionality on the APGE can co-cure with the epoxy and cyanate ester resins. Capote uses this compound, or the difunctional equivalent (2,2'-diallyl bisphenol A diglycidyl ether) in each and every example. Thus, the use of this co-curing agent assures that the epoxy, cyanate ester, and bismaleimide all cure over the same time period, and not in separate stages as in the current invention.

In the cited references, there is nothing to indicate or teach that the individual resins can be cured separately so that the different properties that are present in an uncured adhesive and a cured adhesive can be present simultaneously. At the B-stage in the instant adhesive, the separate cure is the essence of the invention.

Applicants are submitting a second Declaration with this comparing the co-curing invention in Young with the instant invention to further explain and clarify that the instant dual curing composition of this invention is directed to two separate chemistries at two distinct curing temperatures.

Based on the above amendments and remarks, and the enclosed Declaration, applicants urge the Examiner to issue an allowance .

Attorney Docket # 1978.EEM

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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In re application of Kevin Harris Becker      Group Art Unit: 1712

Serial number: 10/016,844      Examiner: SELLERS, Robert E.

Filing date: 14 December 2001      Response date: 28 April 2005

Title: Dual Cure B-Stageable Adhesive for Die Attach

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To:      Commissioner for Patents  
              Alexandria, VA 22313-1450

DECLARATION UNDER 37 CFR 1.132

Sir:      I, Kevin Becker, a citizen of the United States of America, currently residing at 17211 Stowers Avenue, Cerritos, California 90703 declare as follows:

1.      I am an inventor in the instant application and familiar with the issues raised in the Examiner's action on this application.
  
2.      I graduated from Cornell University with a Bachelor of Science degree in Materials Science and Engineering and a Bachelor of Arts in Chemistry in 1989. I received a Ph.D. degree in Materials from the University of California, Santa Barbara, in 1995.
  
3.      Since June 1999 I have worked at Ablestik Laboratories, a subsidiary of National Starch and Chemical Company. I currently hold the position of Business Scientist.
  
4.      My experience has provided me with a strong knowledge of-polymer science and reactive chemistry.
  
5.      The experiments below were conducted under my supervision and guidance to demonstrate that blended compositions as disclosed in US patent **4,816,531** to Young cure at the same temperature and do not anticipate or teach the dual cure chemistry of my invention.

6. A dual cure formulation as claimed in claim 1 of the instant patent application was prepared as follows:

Resin 1 – 30 parts liquid bismaleimide resin, 30 parts monomaleimide resin, 65 parts cinnamyl alcohol / dimer diisocyanate bis-carbamate, and 2.5 parts peroxide initiator (USP-90MD product from Crompton) were mixed together to form Resin 1.

Resin 2 – 40.27 parts rubberized epoxy oligomer, 15.04 parts trifunctional solid epoxy, 1.75 parts dicyandiamide and 0.50 parts amide accelerator were dissolved in 33.39 parts carbitol acetate solvent to form Resin 2 at 63.3% solids by weight.

Resin 1 (11.33 parts) was mixed with Resin 2 (90.95 parts). To this was added 7.5 parts fumed silica and 1.3 parts surfactants to form a B-stageable adhesive.

This formulation is referred to as the Invention Example hereinafter.

7. Limited by the availability of certain raw materials, Young Example 1 was reproduced as closely as possible, as shown in the chart below, and according to the directions as supplied in the Young patent. The choice of substitute materials for those not available is explained in the Comments in paragraph 8 of this Declaration. BMI is bismaleimide and MEK is methyl ethyl ketone. This formulation is referred to as the Young Example hereinafter.

Materials called for in the Young patent example	Substitute Materials used in the Young Example	Description	Amount in parts by weight
Epikote DX-90	Epon Su-8	Bis A novolac epoxy resin	35.3
Epikure DX-175	HRJ-1166	Bis-A novolac resin	13.4
Tetrabromo bisphenol-A	Bis-A 157	Bis-phenol A	3.1
MEK	MEK	Solvent	40
MDA-free hydrazide modified BMI resin	Matrimide A	Bismaleimide resin	60
DMF	DMF	Solvent	40
2-Methylimidazole	2-Methylimidazole	Accelerator	0.24
		Total	192.04
		Solids %	58%

8. Four minor changes were needed. The bis-A novolac epoxy resin (Epikote DX-90) as well as the bis-A novolac resin (Epikure DX-175) used by Young are no longer available, but they were replaced by equivalent resins, Shell Chemical's Epon SU-8 and Schenectedy International's HRJ-1166, respectively. Bisphenol A was substituted for the tetra-bromo analog and the weight percent was adjusted to keep the molar ratio constant (because Ablestik does not allow the use of brominated resins). The BMI used by Young is not commercially available, so a similar solid BMI resin was substituted (CIBA's Matrimide-A). On my information and belief none of these changes should impact the fundamental chemistry behavior of the mixture.

9. Films were prepared from each of the above formulations by casting the formulations (independently) onto 2 mil polyester release liner to a (wet) thickness of about 500um.

The films were then allowed to air dry for the following times:

Invention Example: 2 hours

Young Example: 2 hours

After air-drying, the films were B-staged according to the specified B-stage profiles shown below:

Invention Example: 30 min. @ 90C

Young Example: 2.2 min. @ 163C

After B-staging, the films were cured according to the recommended cure profiles shown below:

Invention Example: 60 min. @ 175C

Young Example: 60 min. @ 180C

The resultant films were all approximately 100um thick after drying.

10. Thermosetting compositions undergo exothermic reactions when the resins contained in the composition undergo curing. Differential Scanning Calorimetry (DSC) can be used to track these reactions and was performed on each film at three different stages: (a) before B-stage, (b) after B-stage, and (c) after cure. The DSC charts for the Invention Example are shown in Figure 1, and the DSC charts for the Young Example are shown in Figure 2, in which the graphs 1(a) and 2(a) are the DSC scans before B-stage, graphs 1(b) and 2(b)

are the DSC scans after B-stage, and graphs 1(c) and 2(c) are the DSC scans after cure.

11. A DSC run before B-stage, that is, before curing, will show a separate exotherm as a (downward sloping) peak in a graph for each reaction that occurs at a particular temperature. If multiple resins participate in the same reaction, there will be only one exotherm. If multiple resins participate in separate curing reactions, there will be multiple exotherms.

12. Figure 1, graph 1(a) shows the DSC peaks for the Invention Example after air drying. The first exotherm, associated with the lower temperature curing resin, has a peak in the range of 150°-160°C and an enthalpy of reaction of 23.6 J/g; the second exotherm, associated with the higher temperature curing resin, occurs in the range of 190°-200°C and has an additional enthalpy of reaction of 23.7 J/g associated with it. This indicates that the BMI and epoxy resins are curing separately. Figure 2, graph 2(a) shows the DSC peaks for the Young Example after air drying. The Young Example exhibits one exotherm between 165°-175°C with an associated enthalpy of reaction of approximately 206 J/g, indicating that both chemistries, BMI and epoxy, are curing at the same time.

13. A DSC run after B-stage should show whether or not both chemistries have cured. If the peak associated with one resin in the system has completely disappeared, and the peak associated with the second resin remains unaffected, this indicates that the first resin has fully cured during B-stage, and the second resin has remained uncured.

14. Figure 1, graph 1(b) shows that only one exotherm remains for the Invention Example after B-stage. The lower temperature peak has completely disappeared indicating that the lower curing resin has completely reacted during B-stage. The second higher curing peak, however, has remained unaffected; it still occurs between 190° -200°C and still has the same enthalpy of reaction (38 J/g, which equals the original enthalpy divided by the solid content, as the solvent is evaporated during B-stage and thus no longer contributes to the weight of the

sample, that is,  $24 \text{ J/g} / 0.63 = 38 \text{ J/g}$ ). This indicates that the cure of the higher curing second resin has not been initiated during B-stage.

15. In contrast, the Young Example was only partially cured. Figure 2, graph 2(b) shows that there is still one peak, but the 147 J/g residual enthalpy of reaction indicates that the total composition has been approximately 25% cured. This indicates that despite the presence of both BMI and epoxy resins, they are co-curing, and that during B-stage **both** resins are being partially cured.

16. The graphs of both the Invention Example, Figure 1, graph 1(c), and the Young Example, Figure 2, graph 2 (c), cure show no peaks after full cure.

17. In my opinion, the above DSC graphs indicate that my invention of a dual curing composition in which two different resins have separate curing events has not been disclosed in the cited prior art.

18. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by a fine or imprisonment or both under 1001 of Title 18 of the United States Code and such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Rancho Dominguez, California, this 26 day of April 2005.



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Kevin Harris Becker, PH.D